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Extreme-temperature lab on a chip for optogalvanic spectroscopy of ultra small samples – key components and a first integration attempt

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Abstract. This is a short summary of the authors' recent R&D on valves, combustors, plasma sources, and pressure and temperature sensors, realized in high-temperature co-fired ceramics, and an account for the first attempt to monolithically integrate them to form a lab on a chip for sample administration, preparation and analysis, as a stage in optogalvanic spectroscopy.

1. Introduction

Despite being widely employed, neither *lab on a chip* (LoC), nor *high temperature* (HT), is really distinctly defined. The latter usually starts already at the “military hot”, i.e., 125°C, and the former covers a really wide range of devices [1]. In fact, LoC could denote just a simple cavity, accessible to the naked eye, or, more often, an elaborate system of channels and chambers, where fluids are controlled and analyzed with auxiliary apparatus, or, more seldom, a rather complicated device with a high degree of functionality containing multiple integrated, active components and feed-throughs.

In this work, extremes in both respects are aimed for by integration of separately developed components into a sophisticated device intended for on-chip sample preparation reaching nearly 1000°C, and analysis, utilizing the optogalvanic effect (OGE) [2].

This LoC is intended for a range of applications, e.g., biomedicine, earth sciences, forensic science, and, ultimately, archeology, all of which benefit from accurate determination of carbon isotope ratios in samples so small and delicate that very little can be wasted on leaks and dead volumes in large instrument assemblies. This calls for a high degree of integration. Reported here, after a short overview of the system and its individual components, is the result from a recent attempt to attain such an integration to form a monolithic, ceramic lab on a chip, from a process perspective.

2. Device Operation and Design

A device like the one conceptualized here is a complex system of passive and active components arranged in line with a certain sequence, here mimicking parts of the macroscopic protocols described



in [3]. Hence, the device under development here, starts with a minute amount of a solid organic sample, turns it into a conductive gas, and analyzes its carbon content with respect to isotope ratio – all on chip. Besides the two components of particularly importance for the sample phase transitions: the *combustor*, gasifying the sample, and the *plasma generator*, making it conductive, to support the OGE, there are valves, sealings, sensors, and channels for sample administration and monitoring.

With reference to figure 1, the sequence of steps from sample insertion to signal acquisition is:

1. insertion of (solid) sample in combustion chamber (to the right)
2. closing of inlet to this chamber (omitted in figure)
3. (gas) evacuation of device through two outlets (middle of rear long side)
4. pressure check through pressure sensors (along front long side)
5. sealing of outlets (omitted in figure)
6. combustion of sample while monitoring temperature and pressure sensor
7. opening of “breakable sealing” (near middle of chip)
8. pressure equalization as monitored through both pressure sensors (same as in (4))
9. temperature stabilization
10. ignition of plasma (in encircled gap of ring-shaped structure to the left)
11. OGE measurement (through wire probes not visible in figure)

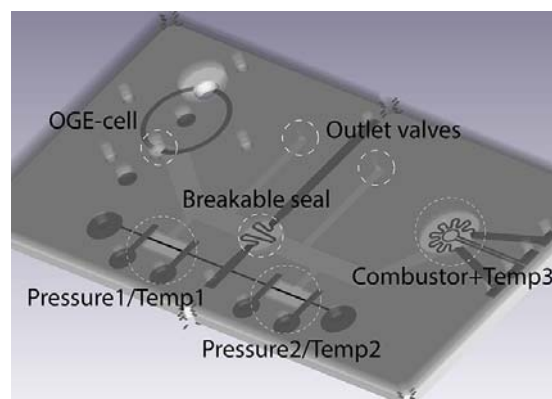


Figure 1. CAD rendering of device, showing all key components, which, in reality, are embedded and invisible. Sample is inserted to the right, signal is read from upper, left corner.

Based on the stacking of separately structured and patterned sheets, it was important to minimize process steps and to avoid accumulation of fabrication errors, such as misalignment, already in the design phase. Figure 2 indicates how embodiment of the above sequence uses vertical and horizontal routing of channels and leads to limit the number of metalized layers to four, and connecting the two ground planes of the stripline plasma source. (In fact, the layers were stepped out in a fashion requiring only one sheet to be metalized.)



Figure 2. Schematic of the device's layers, with grey denoting individual or triplets of ceramic sheets, black metal patterns, green conductive adhesive, yellow an SMA connector, and orange metal wire.

3. Component Overview

With few exceptions, e.g., the innovative use of bond wires and internal electroplating, well-established High-Temperature Co-fired Ceramics (HTCC) technology was used for manufacturing of

all components, both because of the ceramic's good high-temperature properties and its inertness, and because of the coming integration.

The combustor is a resistively heated cavity with the unique feature of having an integrated oxygen supply, formed by oxidizing a copper layer on the heater, and decomposing it by heating. As copper cannot be co-fired with the HTCC alumina, it was selectively electroplated on the platinum heater, subsequently, i.e. inside the cavity. An X-ray image of the final combustor, showing the vias to the heater and temperature sensor leads is shown in figure 3 together with the as-plated copper in an open combustor. Thermogravimetric analysis showed that the oxygen taken up during oxidation was released when heated, starting at around 750°C, and completing at around 900°C. Thermography, again on an open combustor, revealed an increasingly uniform heating of the combustor floor, when the heating power was increased, with a small but noticeable cooling from its leads. Residual gas analysis conducted during the combustion of a carbon-rich sample, together with microscopy, showed that oxygen released from the copper oxide reacted with the carbon, and formed carbon dioxide. [4]

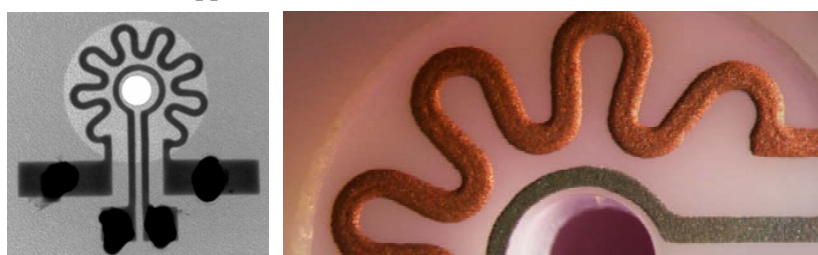


Figure 3. X-ray image of full-fledged combustor with a combined heater and oxygen supply meandering around the circular temperature sensor, inside which the sample inlet and gas outlet combine to make a bright circle (left), and microscope close-up of open reactor just after copper plating of the heater, not having affected the platinum temperature sensor (right).

The plasma source consists of a split-ring resonator, essentially a folded dipole antenna, in this case being sandwiched between ground planes to form a robust and interference-immune stripline device. Where the antenna's two prongs meet, there is a gap through which the sample gas is fed, and over which the electric field is strong enough to ionize it. Following on an extensive development of this kind of plasma sources [5-7], the one used here, was realized with HTCC technology, both for integrational purposes and because of the better thermal and chemical stability of the ceramic. Crucial to this transition, was the investigation of the materials' dielectric properties. From a variation of resonator dimensions, substrate thickness, and metal paste resistivity, it was found that the relative permittivity varied very little, whereas the dielectric loss varied a lot, probably as an effect of a non-uniform metallization thickness. Besides this, important knowledge was gained on how machining of ceramic sheets affects their lamination. In conclusion, a stripline microplasma source with a resonance frequency of 2.6 GHz, producing an intense and stable plasma, was developed. The final stand-alone component is shown in figure 4 together with a close up of the gap during operation. [8]

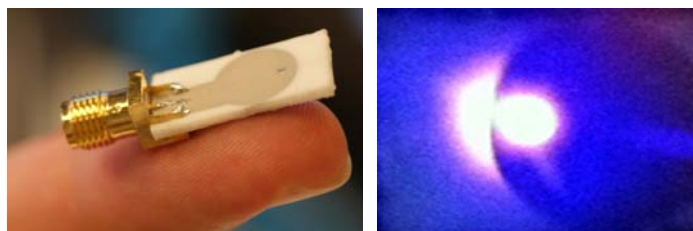


Figure 4. Completed HTCC-based plasma source with one ground plane and the sample and laser beam entrance to the gap of the split-ring resonator visible at top surface (left), and close-up when ignited in air at 10 Torr (right). Because of pixel bleeding and the translucency of the ceramic, the image is blurry and grainy, and the plasma appears much wider than it is.

The system contains two types of single-use valves. Although a second generation of lab chip will probably have a breakable sealing on its inlet to protect its interior from ambient contamination, the version here hasn't, and the first valve encountered in the sequence, is the sealable inlet. This operates by heating and melting a small amount of silver, which, so far, is either electroplated or manually deposited at the rim of the inlet, and having surface tension form a cap. Admittedly, and as evident from figure 5, this part of the device needs further development.

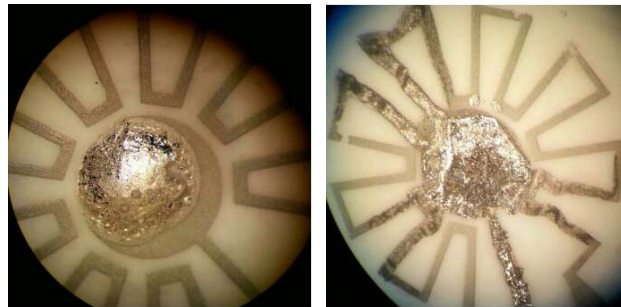


Figure 5. Well made sealing, with, here, manually deposited silver forming a half-sphere over the inlet (left), and the result of minute differences in metal adhesion and/or deposition where the heater meander has started to disadhere from the ceramic, and the molten silver has spread to it from its designated area at the rim of the inlet (right)

The next valve is the opposite, a breakable sealing membrane, which serves to clear the pathway to the analyzer part of the chip. The operation of this is based on causing localized thermal gradients and transients, too large for the brittle ceramic to handle, and controlling crack propagation. To find the right means, a thorough study on heater design, membrane thickness and membrane processing, was conducted. It was found that a few hundred mJ were enough to open most of the thin membranes, and that heater shape and processing scheme played a major role. The resulting openings, ranging from microcracks to loss of the entire membrane, figure 6, were quite reproducible [9].

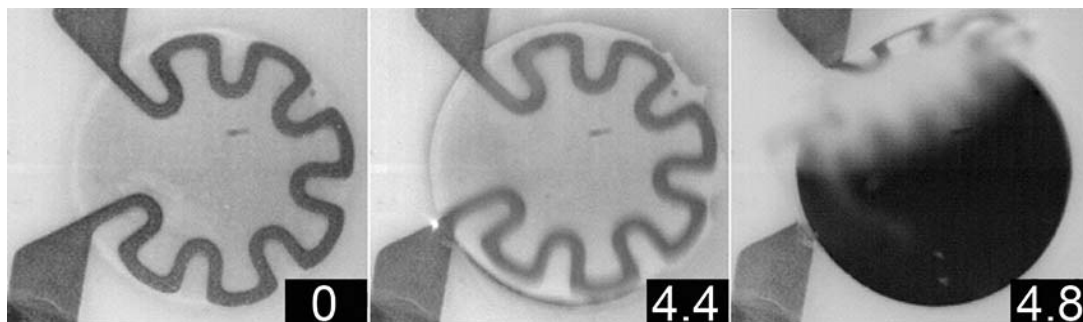


Figure 6. Frames from high-speed footage of a 40 μm thick alumina membrane cracking along its rim as a result of supplying the meander-shaped platinum heater with approx. 350 mJ. The number in each frame's corner is elapsed time in milliseconds from start of heating.

Finally, the chip is equipped with a range of sensors. Besides two plasma probes, there are three temperature sensors and two pressures sensors for housekeeping. Whereas a dedicated temperature sensor, located in the combustor (see figure 3), is made by screen printing platinum, and, hence, following standard HTCC processing, the rest are combined pressure and temperature sensors made by threading platinum bond wires in the stack of ceramic green sheets, prior to sintering. For temperature reading, simply the resistance of such a wire is measured. When heated, however, the wires, raised from their cavity floors as a result from densification of the green sheets on sintering, loose heat convectively in proportion to the surrounding gas pressure, allowing them to work like Pirani gauges, figure 7. Quite remarkably, the performance was satisfactory even on the few occasions the deflection of a wire made it hit the ceiling of its cavity [10].

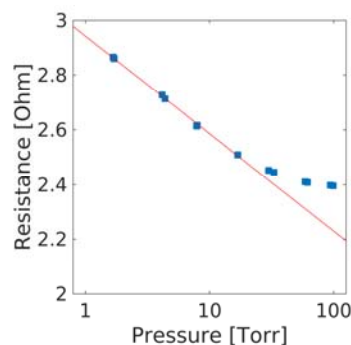


Figure 7. Resistance, being translatable to temperature, of a 25- μm diameter platinum wire vs. pressure in the enclosing cavity

For the plasma probes, which are located in the plasma source's gap, the above wire technology is used, but with a carefully tailored step to fuse them to form rounded tips allowing Langmuir-probe-like measurement of the plasma potential, which is the key to optogalvanic spectroscopy in this embodiment. Compared with the almost spherical tips made previously [11], the tips obtained in HTCC, figure 8, need further work.

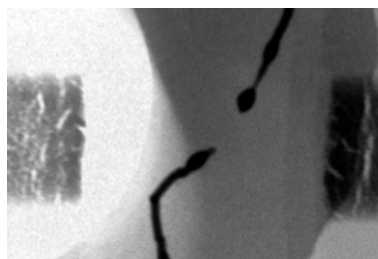


Figure 8. X-ray close-up of two fused plasma probes with semi-spherical tips revealing wire deflection and kinks from sintering.

4. Results at System Level

The integration of all the above components into a 50×25×1.6 mm chip was very successful. Despite the many layers, alignment errors were negligible, figure 9, and the curvature of the whole stack was just a few tenth of a mm. The only actual setback was the unfortunate cutting of the leads just outside the plasma probe cavities (not visible at the figure's magnification). The statistics doesn't allow for any certainty as of the cause, but a reasonable explanation is non-uniform compression of the chip during lamination because of the variation in support between solid green sheet areas and to-be cavities filled with fugitive material. Other than this, all leads and vias followed specifications. Thermography showed that components could be operated individually without severe thermal crosstalk.

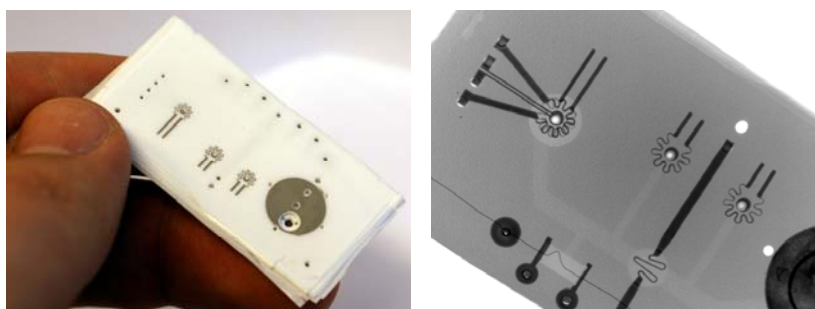


Figure 9. Device after sintering (left), with X-ray close-up of central part, showing alignment and printing quality, and the typical deformation of the sensor wires (right). Darker parts are screen printed platinum or wires, and the lighter shades within the chip are unfilled vias, cavities and channels.

5. Conclusions and Discussion

All inspections and tests conducted, show that integration was successful on the mechanical and thermal grounds defined. Regarding functionality, the damaged probe leads are of course a setback. Changing the locations of the components and routes in-between, may be a solution to this. Near-future work shall entail calibrating the LoC and inserting in the spectroscopy set up, and running the sequence given above. Beyond this, there are both design and process issues deserving further investigation. As for adding features, the above mentioned breakable sealing at the device's inlet is one. Another is a filter to prevent debris from membrane breaking to travel inwards. A third is a pressure reducer, for instance a relatively large cavity lined with getter material and evacuated already during sintering.

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